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- (54) Separation of Bitumen from Tar Sands by Flotation
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# SEPARATION OF BITUMEN FROM TAR SANDS BY FLOTATION

### ABSTRACT OF THE DISCLOSURE

A novel method is provided for improved separation of bitumen from aqueous slurries in a tar sands extraction process by subjecting such slurry or pulp to flotation with a suitable gas such as air or CO<sub>2</sub> in the presence of a solid hydrocarbon, such as coke or coal, as collector.

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# SEPARATION OF BITUMEN FROM TAR SANDS BY FLOTATION

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This invention relates to a flotation method for improved separation of bitumen from aqueous slurries in a tar sands extraction process.

The invention is particularly useful in conjunction with the Hot Water Process for the extraction of bitumen from tar sands, because in this process a number of flotation operations are or can be performed.

Deposits of tar sands, also known as oil sands and bituminous sands, have been identified in many parts of the world including four major deposits in Alberta, Canada.

The Alberta tar sands typically contain between 70 and 90 percent by weight of mineral solids, 5 and 20 per cent of viscous petroleum having a 6° to 10° API gravity and specific gravity of about 1.0 commonly referred to as bitumen, and from about 1 to 10 percent of water. The mineral solids usually referred to as "coarse" are mostly quartz sand over 45 microns in particle size, whereas those referred to as "fines" are mostly clay, silts and fine sands having particle size less than 45 microns. The content of fines has been generally found to increase with the decrease in bitumen content in the tar sands. The usually accepted physical arrangement of the sand-bitumenwater mixture that constitutes the tar sands depicts an



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aqueous phase which envelops the hydrophilic sand grains and separates them from the bitumen phase. The key requirement for the production of bitumen is a fast and complete separation of the bitumen from the mineral solids. Various techniques for separating the bitumen from mined tar sands have been developed to different degrees in the last several decades. These include: direct coking, solvent extraction, cold water separation process, spherical agglomeration and a sand reduction process just to mention a few. These methods are not being used on commercial scale due to a variety of reasons, such as material handling problems, low bitumen recoveries, high energy requirements, solvent losses, etc.

The present commercial plants, Suncor and Syncrude, are based on the Hot Water Process which was initiated in the 1920's by The Research Council of Alberta led by K. A. Clark.

An excellent write-up on tar sands and on bitumen recovery technology is presented by Donald Towson in Kirk-Othmer Encyclopedia of Chemical Technology, Third Edition, pages 602-627.

In the Hot Water Process, the mined tar sands are treated in a tumbler with hot water, steam and additives, usually caustic, to break down the lumps and produce a slurry at approximately 80°C-90°C. The slurry is diluted with hot water to approximately 50% solids, then pumped into a gravity separation cell where entrained air causes the bitumen to float. The flotation process produces the

primary froth, middlings and an underflow, which constitutes the primary tailings.

The secondary froth, which is produced from the middlings by air flotation, contains approximately twice the amount of solids and water as compared to the primary froth. Tailings from the secondary circuit join the primary tailings to form the extraction plant tailings. Following de-aeration and heating, the combined froth stream is further treated by dilution and centrifuging to remove the solids and water from the bitumen in preparation for the upgrading or coking process. The solids and water removed together with residual hydrocarbons constitute the froth treatment plant tailings.

Since the early stages of development of the Hot Water Process, it has been recognized that lower grade ore, with its higher percentage of fines and lower bitumen content, results in lower bitumen recovery in the primary separation stage and increased load (total middlings flow and solids content) in the secondary air flotation circuit. Since the secondary separation produces froth with high, mainly fine solids content, the total solids and water content may surpass the handling capability of the froth treatment plant. Consequently, large amounts of bitumen are lost in the extraction plant tailings. A more efficient separation of bitumen from the middlings stream is highly desirable if a high level of bitumen recovery is to be achieved. This may also allow for reduced cut-off grade of tar sands ore at the mine and increase the total tar sand resource

available for processing.

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Many improvements of the Hot Water Process have been proposed which could be classified under two headings:

- Improved control to increase bitumen recovery and separation efficiency of the existing plants;
- 2. Development of methods for the recovery of the residual hydrocarbons from the tailings and for improving the settling characteristics of the sludge.

Some of these proposals seem to be more effective than others; however, most are not cost effective because they require major modifications to the existing plants.

It is, therefore, the object of the present invention to provide a novel flotation method whereby a high quality froth with high bitumen recoveries is obtained, and this without requiring major modifications to the existing installations. This object is achieved by using a solid hydrocarbon, such as coke or coal, as collector.

It is already known to use solid hydrocarbons, such as coke or coal, to improve bitumen recovery in tar sands operations, as disclosed for example in Canadian Patent No. 1,088,445 of October 28, 1980 by A. Frederick Sirianni and John A. Ripmeester, or in Canadian Patent No. 1,107,216 of August 18, 1981 by William H. Hill. These are agglomeration processes wherein the solid hydrocarbon amalgamates or unites with the bitumen and the so formed

organic phase is then separated from the aqueous phase. In contrast to the above, the present invention relates to the use of a solid hydrocarbon, such as coke or coal as a collector in a flotation method enhancing the collection of bitumen in the form of froth. The solid hydrocarbon collector also acts as a depressant for the minerals by preventing the surface active agents in the pulp to act as a collector for such minerals.

The novel method can be used in any system that utilizes flotation for the extraction of bitumen, but it is particularly useful in conjunction with the Hot Water Process where it can be employed, with advantage, at various stages, such as:

- 1. Tumbler feed
- Middlings stream

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- 3. Tailings stream
- 4. Tailings pond
- 5. Any combination of the above.

In this regard, it should be noted, however, that if

20. the novel process is used in the tumbler feed and/or
middlings stream stages, certain modifications of the extraction and froth treatment plants would be required. On
the other hand, its use at the tailings stream and/or
tailings pond stages would require the construction of a

25 flotation plant with minor modifications to the existing
process.

Thus, according to the present invention, the addition of a solid hydrocarbon as a collector to the tar sands pulp

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to float the bitumen and depress the minerals results in a significant improvement in froth quality (i.e. lower solids content). The solid hydrocarbon may, for example, be coke or coal, which is comminuted to the extent that it can act as collector and it will preferably have a particle size between about 0.07 mm and 1.0 mm. The amount of the solid hydrocarbon to be added may vary depending on the actual process or treatment in which it is employed; however, it will be an amount suitable for the solid hydrocarbon to act as a collector for the liquid hydrocarbon (bitumen) droplets extracted during the process: the most suitable amount can be readily established by those familiar with the art of flotation and, preferably, such collector is used in an amount of 0.1%-6% by weight with reference to the pulp (slurry) on wet basis. In this regard, the collector can be added prior to or during the flotation proper or by stages and, in fact, staged addition of the coke, for example, can decrease the required dosage significantly.

It has also been surprisingly found that the concentration of surface active agents in the pulp is significantly reduced by the use of such collector. This reduction decreases the ability of such surface active agents to act as a collector for the minerals present in the pulp. The minerals are thereby effectively depressed and, thus, the solid hydrocarbon collector also acts as a depressant for the mineral particles.

The flotation can be carried out with air, or with an inert gas such as nitrogen, or with combustion gas if it is

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readily available. However, it has been found that CO<sub>2</sub> is particularly suitable as flotation gas and results in a further increase in separation efficiency, producing higher grade froth.

This process can also be applied for upgrading a typical secondary froth by adding to it solid hydrocarbon as collector and re-floating the same.

Further advantages of the novel method reside in the finding that the tailings from this method can be readily flocculated to produce a faster settling sludge. Also, the presence of the solid hydrocarbon collector in the froth enhances the ease of further separation of the remaining solids from the bitumen. In this regard, it should be pointed out that the amount of solids and water in the recovered bitumen should be as low as possible and the lower the concentration of bitumen in the slurry, the higher the solids recovery for a given bitumen recovery. For example, in the existing process, with bitumen concentration of about 2%-21% in the middlings pulp, the cut-off limit for solids contained in the bitumen phase is usually 2% for a 30% solids slurry. Above this limit, the separation of such solids would present too much difficulty and is unacceptable on industrial scale.

Thus, it is often the solids content (mineral fines) in the recovered bitumen that controls the permissible percentage of bitumen separation itself.

The invention will now be further described with

reference to some non-limitative examples and with reference to the appended drawings in which:

Figure 1 shows comparative values of bitumen recovery versus froth grade;

Figure 2 shows comparative values of bitumen recovery versus froth quality;

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Figure 3 shows comparative values of bitumen recovery versus froth quality on diluted middlings; and

Figure 4 shows comparative curves of solids recoveries and bitumen recoveries versus flotation time.

#### EXAMPLES

1. Tests were conducted on middlings samples which contain 55% wt. solids, 3.6% wt. bitumen and water. 4.5 Kg samples were heated in a flotation cell and kept at  $80^{\circ}$ C. Flotation tests were conducted with air, with  $CO_2$ , with air and collector, and with  $CO_2$  and collector. Collector dosage was 4.5% by weight relative to the slurry and the collector

was 4.5% by weight relative to the slurry and the collector was added at various stages of the flotation process. Froth was collected after 1, 3, 6 and 12 minutes of flotation.

The results for the tests with 210 grams of fluid coke addition into the middlings before flotation are presented in Figures 1 and 2 in comparison with air flotation and CO<sub>2</sub> flotation. By using CO<sub>2</sub> and fluid coke as a collector, the initial recovery of bitumen after 3 minutes increased from 39.5% to 81.9% with froth grade increasing from 18% to 31%, froth grade being defined as bitumen/(bitumen + minerals + water), % wt. in the cumulative froth. The minerals to

bitumen mass ratio in the froth is improved from 1.55 to

- 0.54. Other collector dosages between 0.4% and 5% by weight relative to the slurry were tested in the same manner and gave similar advantageous results.
- 2. Tests conducted on diluted middlings to 30% wt.

  5 solids and 2% wt. bitumen (with other conditions being the same as in Example 1) resulted in similar bitumen recovery rates but with significant improvement in the separation efficiency; mineral to bitumen ratios dropped from 0.62 to 0.39 at the 90% bitumen recovery level, as illustrated in Figure 3.
- 3. Rate tests were conducted on secondary circuit slurry containing 30% solids and 2% bitumen by weight at 80-82°C. The curves in Figure 4 give the results obtained. The two bottom curves indicate results of recovery of solids in the froth without addition to collector (broken curve) and with 4.5% coke addition as collector(solid curve). The two upper curves indicate bitumen recovery under the same circumstances.

It is obvious from this figure that, with a solids cutoff at 2% and without addition of the coke collector, only about 50% of bitumen recovery is achieved, whereas with the addition of the coke collector, about 77% of bitumen recovery is realized, an increase of 54%.

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The above examples clearly demonstrate the effectiveness of the novel method. When applied to the Hot Water
Process plants, the method can be employed mostly with
existing equipment (i.e. flotation circuit) with only minor
process modifications. The fact that coke is readily

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available in such plants as by-product is another significant advantage. The important benefits of the process include higher bitumen recoveries with lower solids content; better control in case of variations in feed stock quality; higher recycle ratio of process waters; reduction in tailings pond requirements and altogether lower cost per barrel of bitumen produced.

The invention is not limited to the specific embodiments described above and any modifications obvious to those skilled in the art are included therein.

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#### CLAIMS

- 1. A flotation method for improved separation of bitumen from aqueous slurries in a tar sands extraction process, which comprises carrying out said flotation in the presence of a solid hydrocarbon as collector.
- 2. A flotation method according to claim 1, wherein the solid hydrocarbon collector is coal.
  - 3. A flotation method according to claim 1, wherein the solid hydrocarbon collector is coke.
- 4. A flotation method according to claims 1, 2 or 3, 10 wherein the solid hydrocarbon collector has a particle size between about 0.07 mm and 1.0 mm.
  - 5. A flotation method according to claims 1, 2 or 3, wherein the solid hydrocarbon collector is used in an amount of about 0.1%-6% by weight with reference to the slurry on wet basis.
    - 6. A flotation method according to claims 1, 2 or 3, wherein the flotation is carried out with the assistance of air.
- 7. A flotation method according to claims 1, 2 or 3 wherein the flotation is carried out with the assistance of CO<sub>2</sub>.
  - 8. A flotation method according to claims 1, 2 or 3, wherein said flotation is carried out with the assistance

of an inert gas, such as nitrogen.

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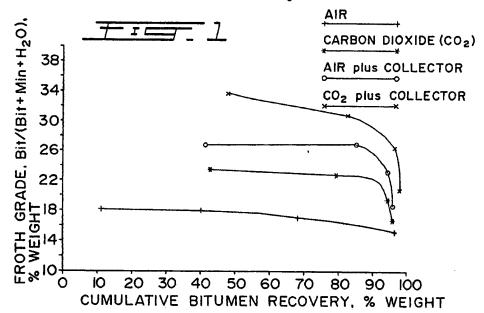
- 9. A flotation method according to claims 1, 2 or 3, wherein the solid hydrocarbon collector is added prior to the flotation proper.
- 5 10. A flotation method according to claims 1, 2 or 3, wherein the solid hydrocarbon collector is added during the flotation.
  - 11. A flotation method according to claims 1, 2 or 3, wherein part of the solid hydrocarbon collector is added prior to the flotation and the remainder during said flotation by stages.
- 12. A flotation method for improved separation of bitumen from aqueous slurries in a tar sands Hot Water Process, which comprises using coke or coal as collector, said collector being added to the slurry in an amount of about 0.1%-6% by weight with reference to said slurry on wet basis, and the flotation being carried out with the assistance of a suitable gas.
- 13. A flotation method according to claim 12,20 wherein said gas is air.
  - 14. A flotation method according to claim 12, wherein said gas is CO2.
  - 15. A flotation method according to claims 12, 13 or 14, which is applied to treat tailings from the pond.

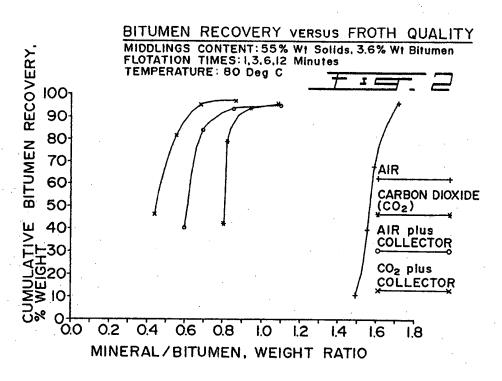
- 16. A flotation method according to claims 12, 13 or
  14, which is applied to treat tailings from the Hot Water
  Process.
- 17. A flotation method according to claims 12, 13 or 14, which is applied to treat the middlings from the Hot Water Process.
  - 18. A flotation method according to claims 12, 13 or 14, which is applied to treat tumbler feed in the Hot Water Process.
- 10. 19. A flotation method according to claims 12, 13 or 14, which is applied to treat a froth from a previous separation in the Hot Water Process.
- 20. A flotation method according to claims 12, 13 or 14, which is applied to treat a plurality of slurries

  from the Hot Water Process.
  - 21. A flotation method according to claims 1 or 12, wherein the solid hydrocarbon collector also acts as depressant for mineral particles present in the slurry.

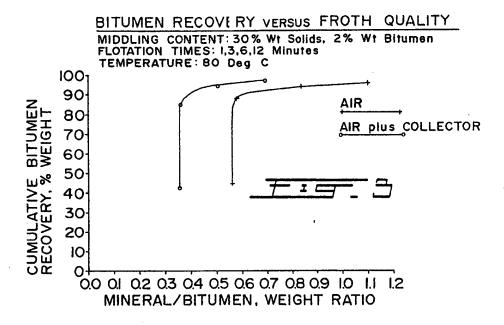
# BITUMEN RECOVERY VERSUS FROTH GRADE MIDDLINGS CONTENT: 55% Wt Solids, 3.6% Wt Bitumen FLOTATION TIMES: 1,3,6,12 Minutes

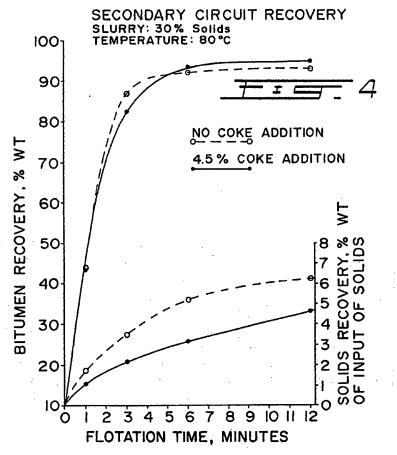
TEMPERATURE: 80 Deg C





Primale & Co.





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